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APPLICANT(S):	ROBERT P. HEPFER, CRAIG T. MILLER, GREGORY A. NORENBERG, THOMAS G. ATTIG, JOHN R. BUDGE	) PATENT) APPLICATION)
APPLICATION NO.:	09/651,526	) GROUP ART UNIT:) 1621)
FILED:	AUGUST 29, 2000	) EXAMINER:) E. PRICE)
FOR:	TWO-STAGE PROCESS FOR THE HYDROGENATION OF MALEIC ACID TO 1,4-BUTANEDIOL	) ATTORNEY DOCKET NO.: 37,241)

CERTIFICATE OF MAILING

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail with sufficient postage in an envelope addressed to: Commissioner of Patents and Trademarks, Washington, D.C. 20231, on or before 9-5-01

Legal Assistant

Date: 9-5-01

DECLARATION UNDER 37 C.F.R. 1.131

We, Robert P. Hepfer, Craig T. Miller, Gregory A. Norenberg, Thomas G. Attig and John R. Budge, do depose and say that:

1. We are the inventors of the claimed subject matter of the above-identified application.

2. We have read U. S. Patent No. 6,008,384 to Bockrath et al.

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3. Our invention is as claimed in the application. However, while not intending to define or limit the invention, the invention may be summarized as a two-stage (i.e. two hydrogenation zones) process for the hydrogenation of maleic acid to 1,4-butanediol (BDO), gamma-butyrolactone (GBL) and/or tetrahydrofuran (THF), wherein the temperature of the first hydrogenation stage or zone is controlled such that the temperature of the maleic acid in the first hydrogenation zone does not exceed about 130°C. Further, temperatures in the second stage or zone is controlled such that the temperature of the reactants in the second hydrogenation zone are between about 130°C to about 180°C.

4. That prior to December 28, 1999, we had completed our invention as described and claimed in the subject application, in the United States, as evidenced by the following exhibits:

(A) Exhibit 1 - Laboratory Notebook No. 17328, Pages 80 and 81

Maleic acid (acquired from a maleic anhydride plant located in Osaka, Japan to which 160 ppm iron was added) was fed to the first of two laboratory scale fixed-bed test reactors connected in series and each containing a catalyst comprising palladium and rhenium on a carbon support. The first reactor (1A) was equipped with two temperature control zones. The second reactor (1B) was equipped with four temperature control zones. The effluent from the second reactor comprised BDO, GBL and THF.

The temperatures for each zone are listed in the Table near the top of page 80 which is duplicated below:

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TEMPERATURES

	1A		1B	
	Set	Internal	Set	Internal
Zone 1	-	79.4	165	157.7
Zone 2	-	106.5	166	171.0
Zone 3	125	124.1	175	172.9
Zone 4	135	130.4	175	177.7

Reactor temperatures employed in reactor 1A (i.e. the first hydrogenation zone) did not exceed about 130°C. Reactor temperatures employed in reactor 1B (i.e. the second hydrogenation zone) were between about 130°C to about 180°C.

Reactor Effluent from reactor 1B was analyzed using gas chromatography, the chromatograph printouts are shown on the bottom of page 80. The product slate as calculated is shown on page 81.

(B) Exhibit 2 - Table 5 from Topical Report No. 8697

A Topical Report is a very detailed internal report of research work completed as well as any findings or conclusions resulting from the research. The reports are signed and dated by their authors and are made available to other researchers, managers and other interested parties within the organization. The author of Topical Report No. 8697 was John R. Budge.

Exhibit 2 illustrates "Time-on-Stream" Data for a simulated two-stage process for the conversion of maleic acid to BDO, GBL and THF. For these experiments a single 200 cc fixed-bed test reactor was utilized (i.e.

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the same reactor was used for the "first stage" and the "second stage", but the stages were not conducted simultaneously).

The two-stage process was "simulated" by conducting in the reactor, the "first stage" reaction of maleic acid to succinic acid at reaction temperatures less than about 130°C, and then later conducting in the reactor, the "second stage" reaction of succinic acid to BDO, GBL and THF at temperatures between about 130°C and about 180°C. Both stages employed a catalyst comprising palladium and rhenium on a carbon support. However, different catalysts were employed for each stage. The succinic acid feed for the second stage was freshly prepared succinic acid and not the reaction product of the "first stage" simulation.

Table 5A represents the first stage (i.e. first hydrogenation zone) data where it can be seen that the average temperatures did not exceed about 130°C. Tables 5B, 5C (labeled as "Comparison of Second Stage Simulation in 40cc and 200cc Reactors") and 5D represent the second stage (i.e. second hydrogenation zone) data where it can be seen that the average reactor temperatures were between about 130°C and about180°C.

This combination of experiments helped to establish the viability of the temperature parameters for hydrogenation zones as claimed in the above identified application.

The experimentation supporting the above exhibits were done personally by Thomas G. Attig and/or John R. Budge or by others under the direction of Thomas G. Attig and/or John R. Budge

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The Exhibits are true copies of actual Laboratory or Topical Report pages, except that the dates thereof have been blanked out. The dates of these documents and all supporting work are prior to December 28, 1999.

5. The undersigned declare further that all statements made herein of their own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

ROBERT P. HEPFER — 8/23/01 Date

CRAIG T. MILLER — 8/28/01 Date

GREGORY A. NORENBERG — 08/23/01 Date

THOMAS G. ATTIG — 8/23/01 Date

JOHN R. BUDGE — 8/29/01 Date

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